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(54) **A Process for Recovering Triglycerides**

(57) A process is disclosed for recovering triglycerides or mixtures of triglycerides of higher fatty acids in crystalline form from naturally occurring fats and oils which comprises heating the fat and/or to dissolve all the crystal nuclei, rapidly

cooling the melt to a temperature 5 to 15°C above its solidification point and adding seed crystals to the melt maintained at that temperature, then slowly cooling the melt to a temperature in the range from 4°C above to 4°C below its solidification point and recovering the crystals formed. Large, pure crystals of the required triglyceride or triglyceride fraction are obtained in this way.

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## SPECIFICATION

## A Process for Recovering Triglycerides

This invention relates to a process for recovering triglycerides and, more particularly, to a process for recovering triglycerides or mixtures of triglycerides of higher fatty acids in crystalline form from naturally occurring fats and oils.

For some time now, the recovering of natural fats in highly pure form has been acquiring increasing significance. This is primarily attributable to the fact that certain triglycerides of high fatty acids or certain mixtures of triglycerides of the type present in natural fats and having selected properties, such as clearly defined melting points or melting point ranges, degrees of unsaturation, rheological behaviour and the like, are desirable for various purposes. Natural fats are generally mixtures of various triglycerides in which glycerine is differently esterified with a variety of fatty acids. As a rule, all these triglycerides have relatively closely related properties so that they are very difficult to separate by techniques such as distillation. In addition, these triglycerides, particularly when they contain unsaturated fatty acids in the molecule, are highly sensitive to the effect of oxygen, particularly at elevated temperatures such as have to be applied during distillation. For this reason, efforts have been made to find other separation processes for separating the individual triglycerides in the natural fats from one another or for isolating particular mixtures of individual triglycerides from the natural fats.

Various natural fats or individual triglycerides of higher fatty acids are known to be polymorphous, i.e. they occur or are capable of occurring in various crystal forms. Thus, numerous fats are known to have  $\alpha$ -,  $\beta$ - and  $\beta'$ -forms. These various crystal forms have different properties which are not only confined to the position of the molecules relative to one another. It is also known that the individual crystal forms can be transformed at certain temperatures, the so-called transformation temperatures, and thereafter shown another crystal form.

This knowledge formed the basis of the present invention of which the object is to provide a process for recovering pure triglycerides of higher fatty acids or certain fractions of triglycerides of higher fatty acids in crystalline form.

Accordingly, the present invention provides a process for recovering triglycerides, or mixtures of triglycerides, of higher fatty acids in crystalline form from naturally occurring fats and oils wherein

(a) naturally occurring fats and/or oils are heated to a temperature above the nucleus formation temperature and all the crystal nuclei are dissolved,

(b) the melt is then rapidly cooled to a temperature 5 to 15°C above the solidification point,

(c) seed crystals optionally suspended in a small quantity of the naturally occurring oil and/or

molten fat used are added to the melt at that temperature,

(d) the melt containing the added seed crystals is cooled slowly to a temperature from 4°C above to 4°C below the solidification point and

(e) the crystals formed at that temperature are recovered.

In the first stage (a), the fat and/or oil is briefly heated to a temperature above the nucleus formation temperature, provision being made to ensure that all the crystal nuclei dissolve, i.e. to ensure that the melt is free from nuclei. This temperature is generally above 75°C and, more particularly, in the range from 80 to 90°C. It is preferably reached particularly economically using a heat exchanger with peak heating.

When the melt leaves the heat exchanger, its temperature is 5 to 15°C and preferably 8 to 12°C above the solidification point. When the melt has reached this temperature, seed crystals, preferably in the  $\beta$ -crystal form, are added in a quantity of preferably from 1 to 100 g per tonne of fat and/or oil. The seed crystals may be suspended in the oil or molten fat used and may be added to the melt in this form. In this way, only a very small quantity of oil and/or fat is required.

After the seed crystals have been added, the temperature of the melt is slowly reduced until it is within the range between 4°C above and 4°C below the solidification point. Thorough mixing of the melt is particularly advantageous at this stage. The solidification point and, hence, the cooling rate of the melt vary according to the required triglyceride or mixture of triglycerides of high fatty acids. In order to crystallise the melt, it is best to use a crystalliser of which the cooling surface is kept at a constant temperature. This is done by means of a coolant of which the temperature corresponds to the filtration temperature.

The temperature lying within the range from 4°C above to 4°C below the solidification point is also known as the filtration temperature. When the melt has reached the filtration temperature, filtration of the crystals formed is commenced.

The filtration obtained after filtration may then be subjected to another crystallisation process of the type described above. In that case, the necessary temperatures for the other triglycerides or triglyceride mixtures required are selected for crystallisation and filtration.

The effect of the process according to the invention is that the triglycerides crystallise in pure form, in other words they are formed without the inclusion of parts of the melt or other molecules. In addition, it is possible for the crystals to become very large because, the larger the crystals, the smaller their specific surface and hence the smaller the danger of impurities being entrained by wetting of the surface during separation of the crystals. In addition, crystals which are fairly large in size are easier to separate off from the melt, for example, by filtration.

The melt must not be undercooled in the process according to the invention. On the

contrary, crystallisation has to take place slowly because it is only in this way that the required crystals are selectively formed without unwanted molecules being incorporated into the crystal lattice.

Large, highly pure crystals of the required triglyceride or triglyceride mixture are obtained in this way.

- In view of the fact that, with natural fats, the crystallisation process is very slow, it was surprising to find that the crystallisation times can be shortened by virtue of the fact that it is only at those temperatures at which significant quantities of crystals are formed that a very gradual reduction in temperature need be carried out up to the point at which crystal formation virtually stops, whereas the other process steps may be carried out fairly quickly. In this way, the total time normally required for crystallising fats is considerably shortened and the triglycerides are kept largely intact.

Control of the temperature at which significant quantities of crystals are formed is particularly important and requires careful monitoring. In this connection, it has proved to be advantageous to follow the trend of the temperature in the melt by means of a temperature recorder.

The invention is illustrated by the following Examples.

### 30 Example 1

15 kg of de-acidified and bleached palm oil were heated to 85°C, all the crystal nuclei dissolving. The melt was then rapidly cooled to 45°C. At this temperature, 1 g of seed crystals suspended in palm oil at temperature of 45°C was added to the melt. The melt was then slowly cooled to 35°C which took about 2 to 3 hours. The crystals formed are filtered off. Yield: 2.5 kg; iodine number: 31; m.p.: 54°C.

- 40 12.5 kg of olein as filtrate were returned to the crystalliser where seed crystals were added. The cooling temperature was then adjusted to 20°C. When the melt reached this temperature, which was the case after 2 to 4 hours, the crystals formed were filtered off. Yield: 6.5 kg; iodine number: 33; m.p.: 29°C.

An olein (6.0 kg) having an iodine number of 60 and a turbidity point of 5°C was obtained as residue.

### 50 Example 2

750 g of refined edible tallow (premier jus) having an iodine number of 44.9 and a solidification point of 36.0°C were heated to 85°C, rapidly cooled with stirring to 46°C and  $\beta$ -tristearin seed crystals were added at that temperature. The fat was then placed in a temperature bath at 35°C and further stirred. After reaching this temperature the fat was left for another 2 hours to crystallise. The crystal sludge formed was then filtered, giving:

1. Stearin: 406.7 g (= 54.2%) with an iodine number of 40.3, a melting point of 46.7°C

and an (extrapolated) refractive index  $n_D^{40}$  of 1.4566.

- 65 2. Olein: 343.3 g (= 45.8%) with an iodine number of 50.4, a melting point of 29.8°C and a refractive index  $n_D^{40}$  of 1.4580.

### Example 3

- 750 g of hardened soya bean oil having an iodine number of 77.0 and a solidification point of 31.5°C were heated to 85°C and then rapidly cooled with stirring to 43°C, followed by the addition at that temperature of  $\beta$ -tristearin seed crystals. The fat was then placed in a temperature bath at 32°C and further stirred. After reaching this temperature, the fat was left for another 2 hours to crystallise, giving:

1. Stearin: 331.7 g (= 44.3%) with an iodine number of 73.2, a melting point of 38.2°C and a refractive index  $n_D^{40}$  of 1.4599.
- 80 2. Olein: 418.3 g (= 55.8%) with an iodine number of 80.0, a melting point of 29.9°C and a refractive index  $n_D^{40}$  of 1.4608.

### Example 4

- 750 g of hardened fish oil having an iodine number of 75.6 and a solidification point of 32.2°C were heated to 85°C and rapidly cooled with stirring to 43°C, followed by the addition at that temperature of  $\beta$ -tristearin seed crystals. The fat was then placed in a temperature bath at 32°C and further stirred. After reaching this temperature, the fat was left for another 2 hours to crystallise, giving:

- 95 1. Stearin: 421.8 g (= 56.2%) with an iodine number of 71.9, a melting point of 38.1°C and a refractive index  $n_D^{40}$  of 1.4600.
2. Olein: 328.2 g (= 43.8%) with an iodine number of 80.3, a melting point of 31.2°C and a refractive index  $n_D^{40}$  of 1.4609.

- 100 The melting points quoted in the preceding Examples were so-called "STEIG" melting points which, like the solidification points, were measured by the standard DGF method, Abt. C-Fette, CIV3a(52).

### 105 Claims

1. A process for recovering a triglyceride of a higher fatty acid, or a mixture of such triglycerides, in crystalline form from a naturally occurring fat and/or oil wherein

- 110 (a) a naturally occurring fat and/or oil is heated to a temperature above the nucleus formation temperature such that all the crystal nuclei are dissolved,

(b) the resulting melt is rapidly cooled to a temperature 5 to 15°C above the solidification point,

- 115 (c) seed crystals optionally suspended in a small quantity of the naturally occurring oil and/or molten fat used are added to the melt at that temperature,

(d) The melt containing the added seed crystals is cooled slowly to a temperature from 4°C above to 4°C below the solidification point, and

(e) the crystals formed at that temperature are recovered.

2. A process as claimed in Claim 1, wherein in stage (b), the melt free from crystal nuclei is cooled to a temperature 8 to 12°C above the solidification point.
- 5 3. A process as claimed in Claim 1 or 2, wherein in stage (c), seed crystals are added in a quantity of from 1 to 100 g per tonne of fat and/or oil.
- 10 4. A process as claimed in any of Claims 1 to 3, wherein in the seed crystals are  $\beta$ -seed crystals.

5. A process as claimed in any of Claims 1 to 4, wherein the melt is thoroughly mixed during crystallisation.
- 15 6. A process as claimed in any of Claims 1 to 5, wherein stages (a) to (e) of the process are carried out with the filtrate obtained after filtration of the first crystal fraction at the temperatures required for the following crystal fraction.
- 20 7. A process as claimed in claim 1, substantially as herein described with reference to any of the specific Examples.

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